

Thermal Desorption Spectroscopy studies of hydrogen retention on FLIRE

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Outline

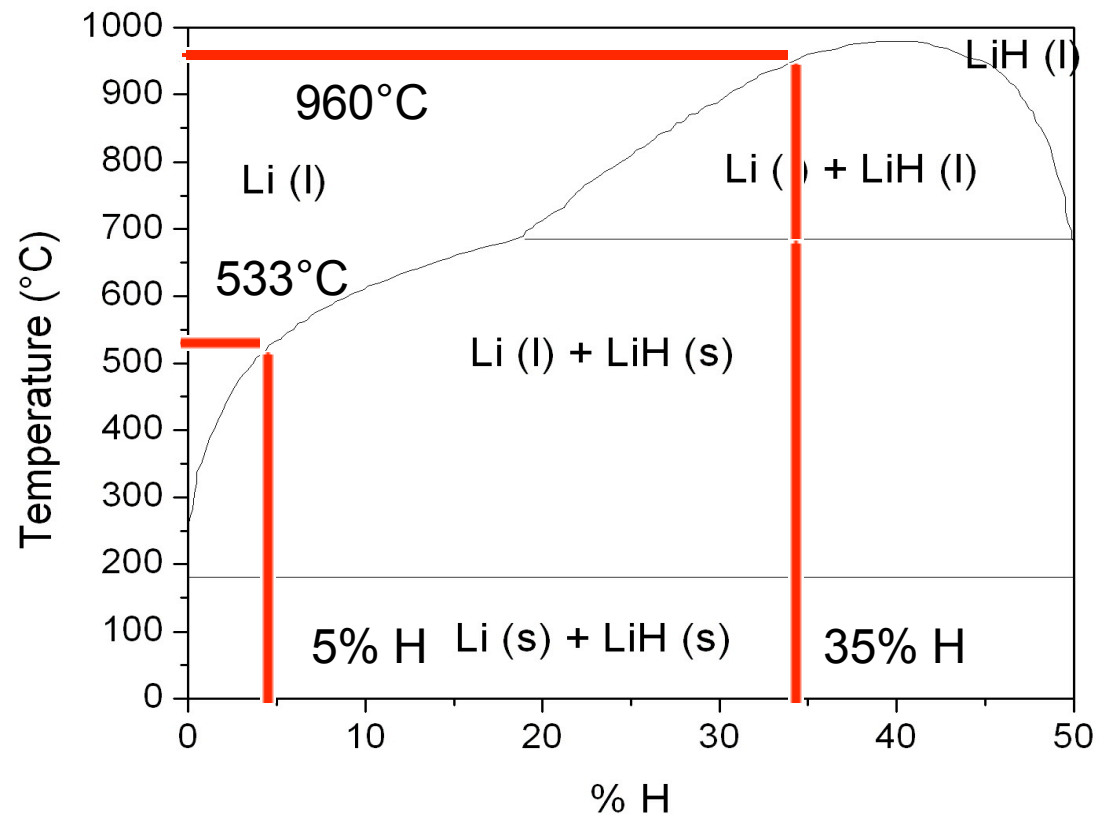
- Introduction
- Thermal Desorption Spectroscopy (TDS) system description
- Experimental results
- Conclusions/Future work

Importance of H retention measurements

- We need to find out how much hydrogen will be carried away by a liquid PFC
- Tritium inventory may be unacceptable
- Low recycling regime may not be achieved

Solubility of H in Li

- Unlike He, H may be trapped in the Li for a longer time
- Hydrogen is soluble in lithium
- Hydride formation is very likely.
- Below 19% no liquid LiH is formed



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* Data taken from: The chemistry of the liquid alkali metals, C.C. Addison. John Wiley & Sons. 1984.



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Need for a Thermal Desorption Spectroscopy (TDS) system

- Quantification of hydrogen content – how much is actually retained in the flowing liquid Li
- Study of hydrogen desorption mechanisms from lithium surfaces
- Higher temperatures can be achieved in the TDS chamber than anywhere else on FLIRE

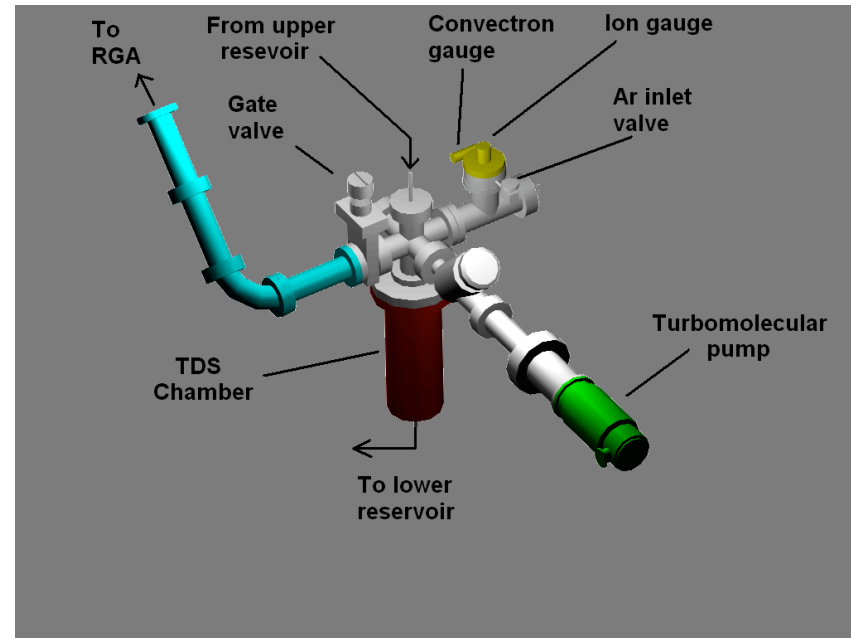
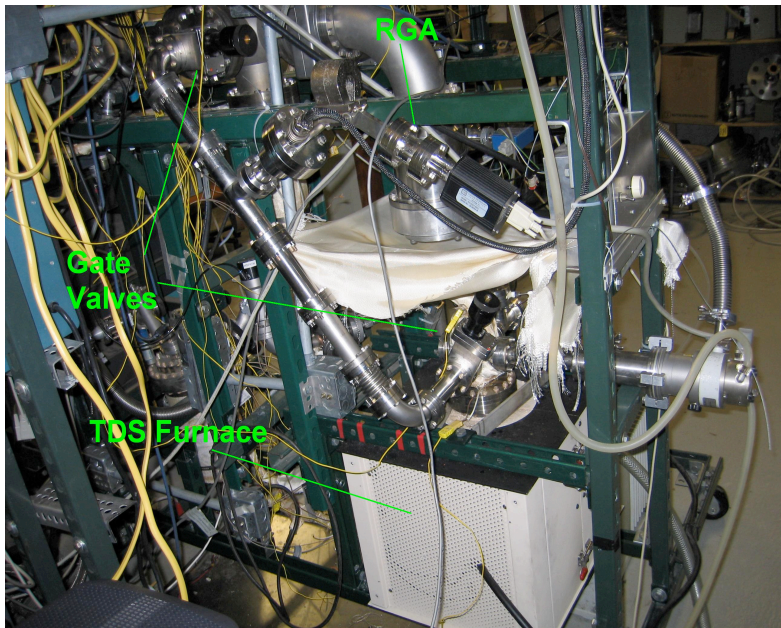


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The Thermal Desorption Spectroscopy (TDS)



- Small TMP evacuates the TDS chamber
- RGA shared with the lower chamber
- PID controller can ramp up TDS temperature at a constant rate

Experimental conditions

- Flow parameters
 - γ Lithium mass ~ 400 g
 - γ Flow velocity of 60 cm/s for 26 sec
 - γ One ramp only at 230 °C
- First run
 - γ No deuterium exposure
 - γ Thermal treatment repeated after TDS was drained
- Second run
 - γ Lithium flow exposed to neutral deuterium gas at 6.0×10^{-5} Torr
- TDS experiment
 - γ Heated from 250 to 600 °C in ~ 3 hrs (2 °C/min)
 - γ Soaked at 600 °C for 1 hour
 - γ Natural cool down

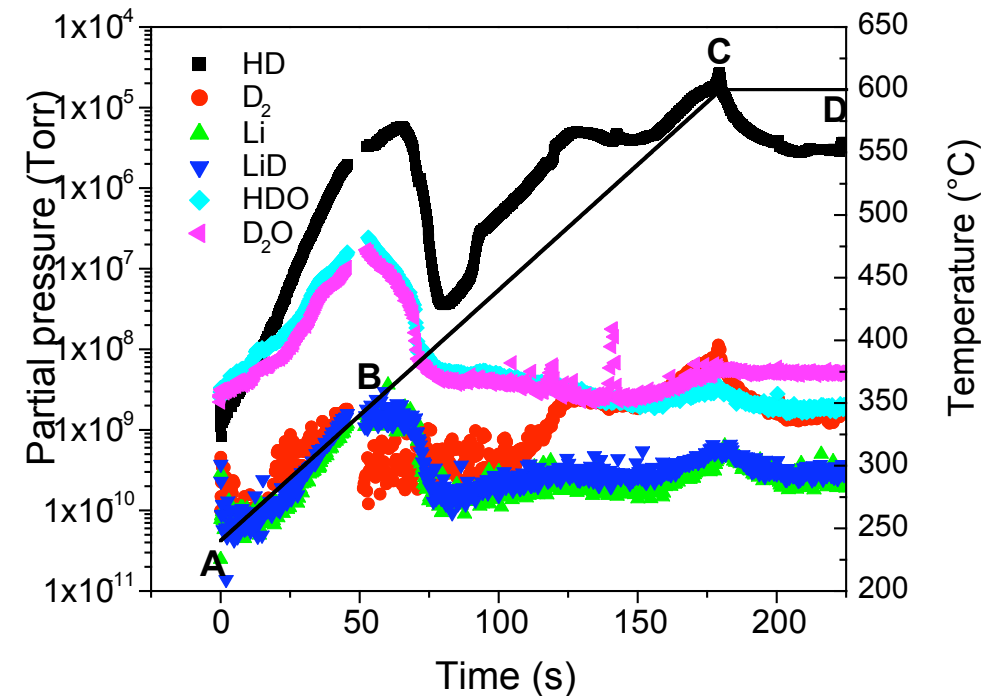


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Initial TDS run with Li and Ion Gun

- Desorption spikes at 350 °C
- Spike is associated with phase change
- Corresponds to about 0.7% hydrogen
- High water peaks may indicate exaggerated hydrogen content



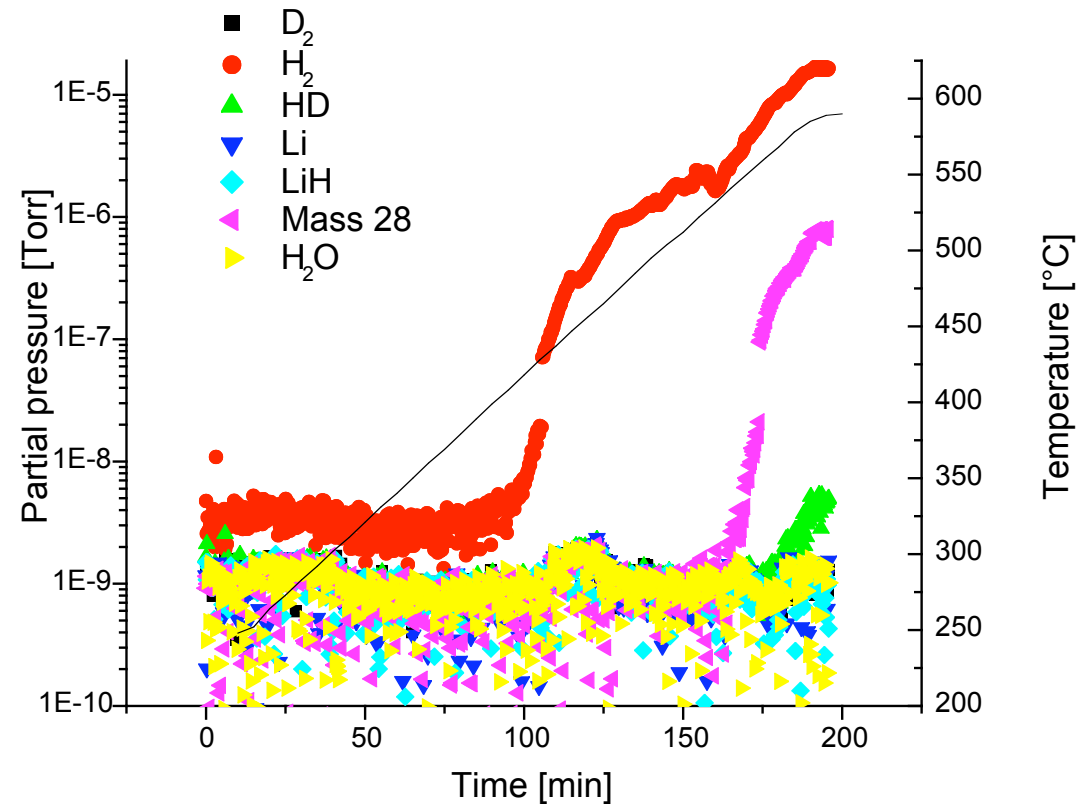
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Li in TDS without D₂ treatment

- No desorption spike at 350 °C
- No water peaks
- No lithium peaks

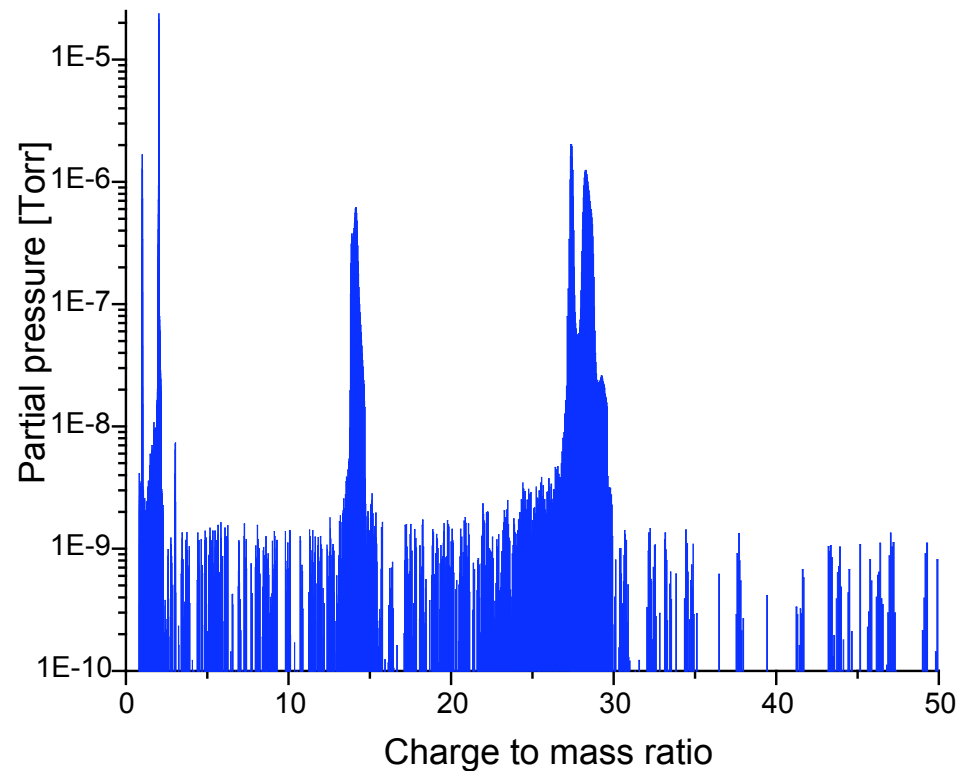


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Mass scan during 600 °C plateau

- Mass 14 and 28 peaks might indicate leak at high temperature (CO and/or N₂)
- Oxygen must be consumed by lithium



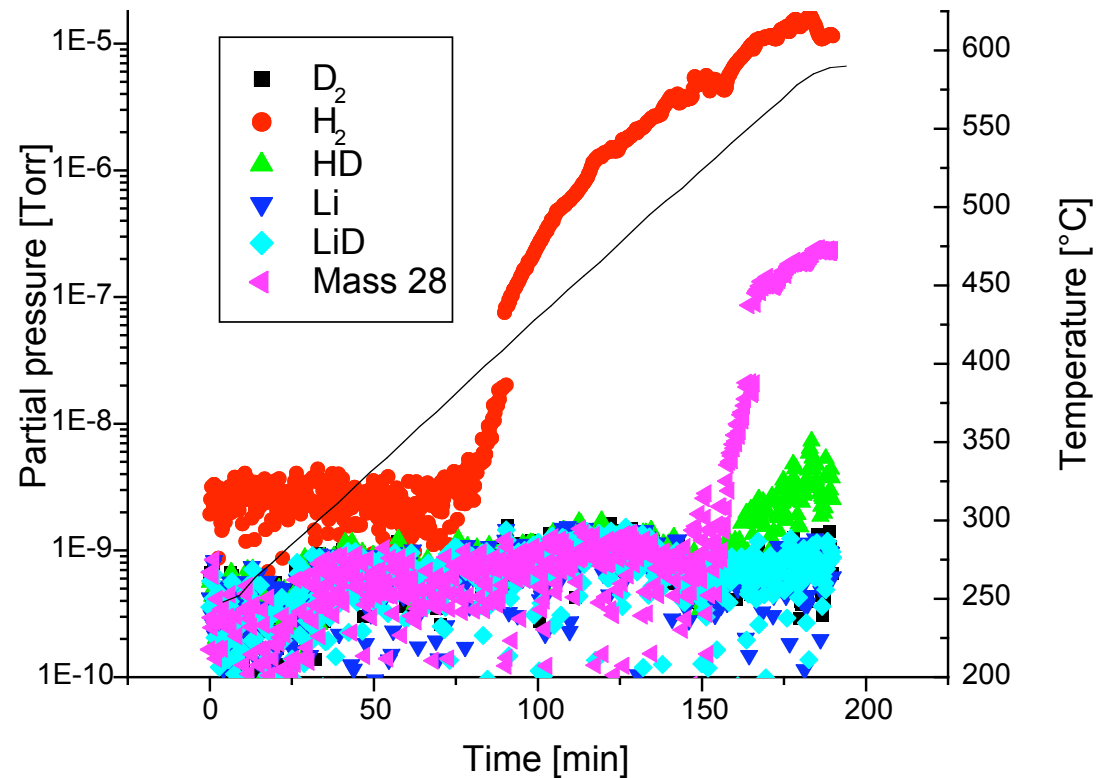
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TDS Drained (no Li)

- Very similar to TDS with unexposed lithium
- Desorption from adjacent surfaces starts at $\sim 400^\circ\text{C}$
- Sudden increase of Nitrogen at $\sim 550^\circ\text{C}$

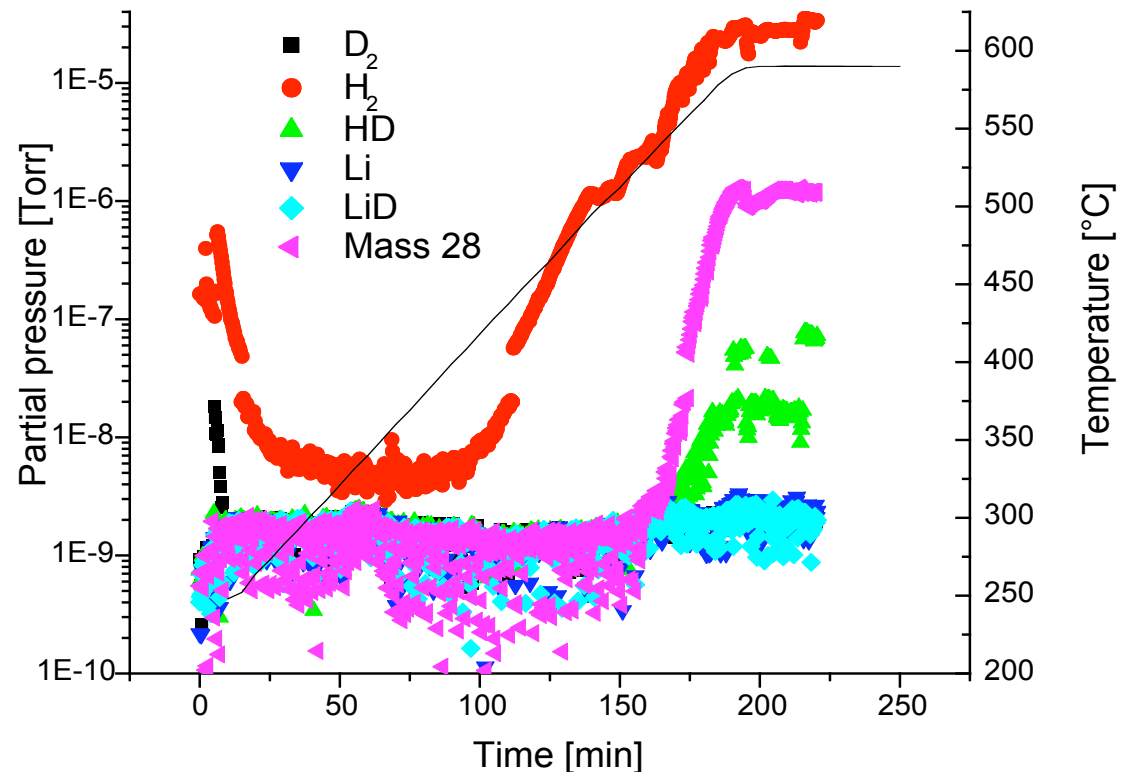


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Li exposed to neutral D₂

- Hydrogen and deuterium peak at low temperature
- Higher-temperatures are unchanged

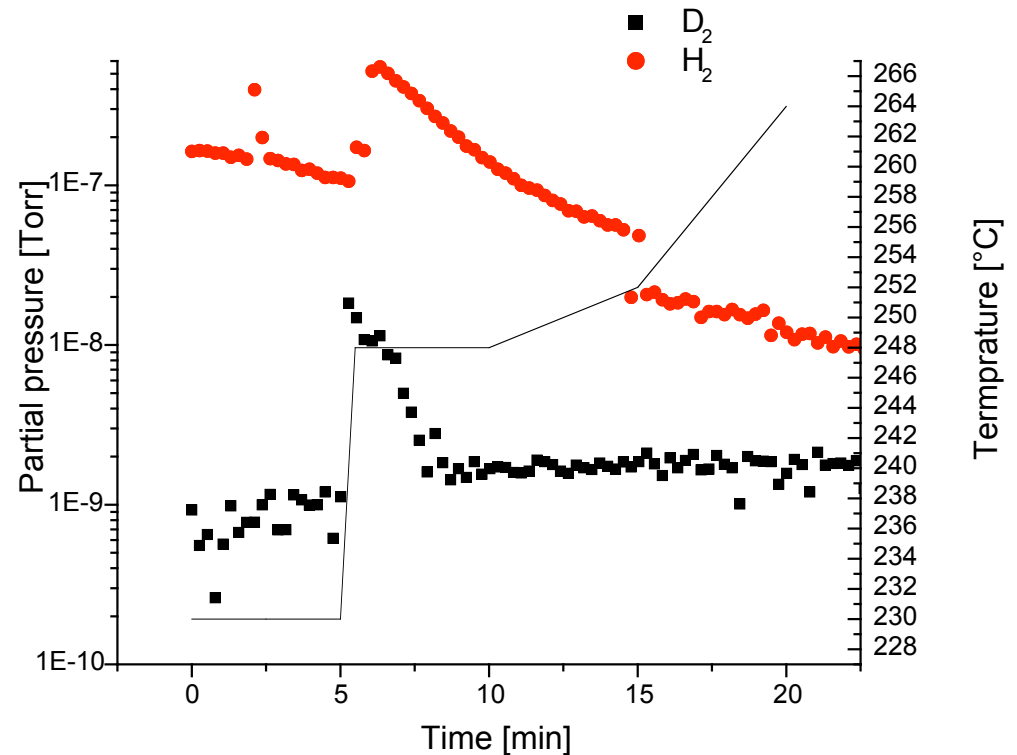


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First 30 min of the run

- Transferred to TDS chamber 5 minutes after scan was started
- Temperature difference, $\sim 20^\circ\text{C}$, results in immediate H_2 and D_2 spikes
- Low temperature desorption indicates lower H_2 and D_2 concentration, 0.1-0.2%



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Results

- No Lithium vapor was observed (condensation)
- Exposure of Li to 6×10^{-5} of neutral D_2 gas resulted in less than 0.5% of LiD concentration
- Most of the desorption occurred even before heating of the chamber started.
- Increased N_2 pressure was observed at higher temperatures ($\sim 550^\circ C$), which is likely caused by micro-leaks due to thermal stresses on the gaskets



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Conclusion

- Initial absorption-tower model over-estimates hydrogen uptake, ~3%, because dissociation not taken into account
- Lithium absorption is 0.1-0.2% for 6×10^{-5} Torr of exposure
- Exposure with ion gun in initial runs indicates ~0.7% absorption
 - γ Water content may have exaggerated hydrogen concentration
 - γ More data with better cleaning/conditioning procedures needed
- Higher-temperature TDS trace is due to external surfaces – empty tank trace nearly identical to non-exposed tank of lithium



Future work

- Irradiate lithium stream under various conditions (Energy, Ion current, flow velocity, pressure, etc.)
- Quantify the amount of D trapped in the liquid Li as a function of flow parameters
- Test heat treated low carbon steel gaskets for improved high temperature vacuum seal



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Hardness test

- Copper = 8.1 HRB
- 1010 Steel original = 96.5 HRB
- 1010 Steel annealed at 800 °C for an hour = 46.4 HRB



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